

# **DETERMINATION OF PAHs IN WATER SAMPLES**

**CARLOS ADELANTADO SÁNCHEZ**

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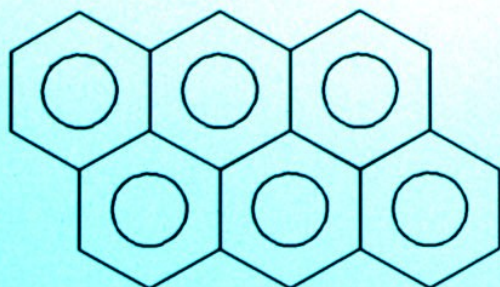
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- Definition of PAHs.
- Structure.
- Features.
- Analysis by SPE-GC-FID.
- Analysis by GC-MS-SIM.

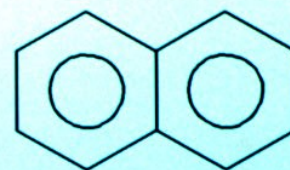
# WHAT IS A PAH?

Polycyclic Aromatic Hydrocarbons (PAHs) are a group of chemicals that occur naturally in coal, crude oil and gasoline. PAHs also are present in products made from fossil fuels, such as coal-tar pitch, creosote and asphalt. When coal is converted to natural gas, PAHs can be released. Therefore, some former coal-gasification sites may have elevated levels of PAHs. PAHs also can be released into the air during the incomplete burning of fossil fuels and garbage. The less efficient the burning process, the more PAHs are given off. Forest fires and volcanoes can produce PAHs naturally.

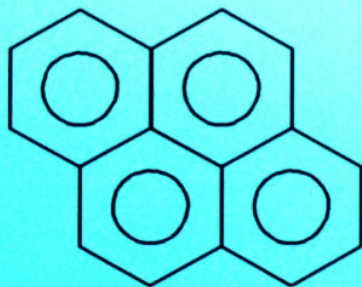
# STRUCTURE



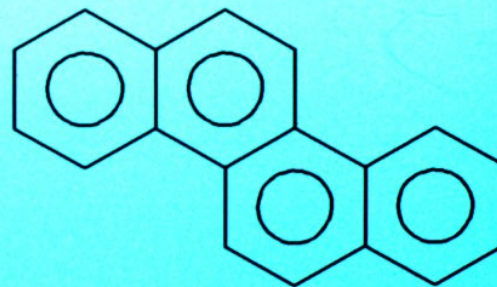
**Anthanthrene**  
 $C_{22}H_{12}$



**Naphthalene**  
 $C_{10}H_8$



**Pyrene**  
 $C_{16}H_{10}$



**Chrysene**  
 $C_{18}H_{12}$

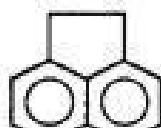


# STRUCTURE

*S. C. Wilson, K. C. Jones*



Naphthalene  
(C<sub>10</sub>H<sub>8</sub>) \*



Acenaphthene  
(C<sub>12</sub>H<sub>10</sub>) \*



Acenaphthylene  
(C<sub>12</sub>H<sub>8</sub>) \*



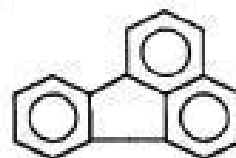
Fluorene  
(C<sub>13</sub>H<sub>10</sub>) \*



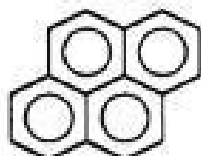
Phenanthrene  
(C<sub>14</sub>H<sub>10</sub>) \*



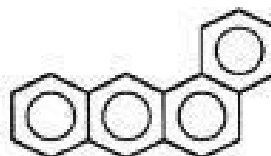
Anthracene (C<sub>14</sub>H<sub>10</sub>) \*



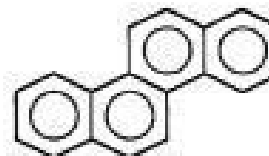
Fluoranthene (C<sub>16</sub>H<sub>12</sub>) \*



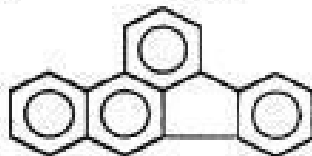
Pyrene  
(C<sub>16</sub>H<sub>10</sub>) \*



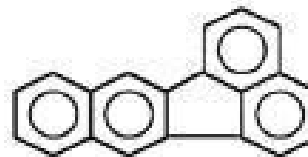
Benz(a)anthracene  
(C<sub>18</sub>H<sub>12</sub>) \*



Chrysene (C<sub>18</sub>H<sub>12</sub>) \*



Benzo[k]fluoranthene (C<sub>20</sub>H<sub>12</sub>) \*



Benzo[k]fluoranthene (C<sub>20</sub>H<sub>12</sub>) \*

# FEATURES

Physical and chemical characteristics of PAHs vary with molecular weight. For instance, PAH resistance to oxidation, reduction, and vapourization increases with increasing molecular weight, whereas the aqueous solubility of these compounds decreases. PAHs can be divided into two groups based on their physical, chemical, and biological characteristics. The lower molecular weight PAHs (e.g., 2 to 3 ring group of PAHs such as naphthalenes, fluorenes, phenanthrenes, and anthracenes) have significant acute toxicity to aquatic organisms, whereas the high molecular weight PAHs, 4 to 7 ring (from chrysenes to coronenes) do not.

# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

## Sample:

- Contains naphthalene (around 4 ppm).
- Take 500 mL.
- Add 5 mL of isopropanol.
- Add the surrogate in the same amount of the analyte.

## SPE cartridge:

- Type: C<sub>18</sub>.
- Fit out with 10 mL of isopropanol.
- Load the sample in the cartridge.
- Dry the cartridge by the exposure to the environment.
- Elute the sample with 2-3 mL of acetone/ethyl acetate (75:25).
- Collect in a 10-mL flask, then add the internal standard.

# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

## Standards:

- Calculate the concentration of naphthalene and prepare 4-5 standards in such a way that the sample is in the middle.
- In each standard there must be naphthalene, biphenyl and benzophenone.

## Chromatographic program:

- Prepare a program in which the three analytes are separated.

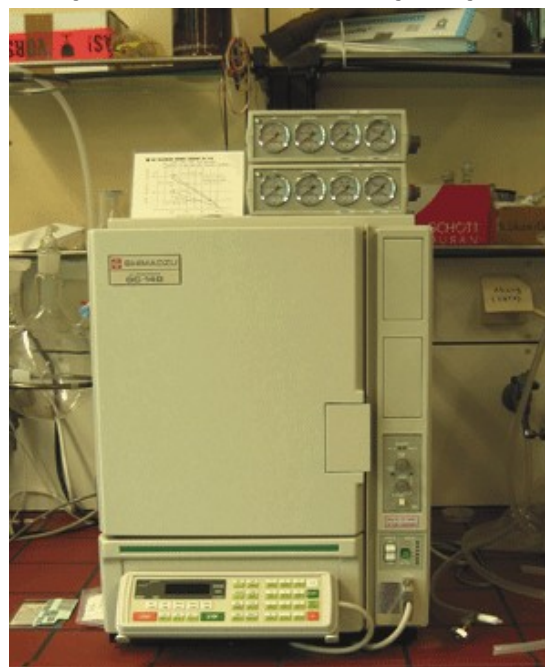
## Analysis:

- Inject the standards and the sample.
- Calculate the concentration of naphthalene in the sample.
- Calculate the recovery of the method.

# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

## Equipment:

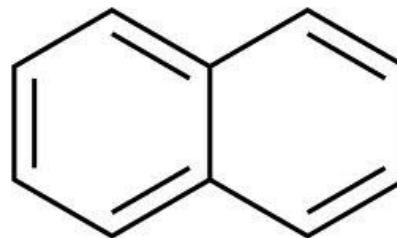
- Gas Chromatograph GC 14-B Shimadzu (Sh 14).
- Column: TBR-5 (30 m x 0.25 id x 0.25 $\mu$ m sp).
- Stationary phase: DB-5 (5% phenyl–95%methylsyloxane).
- Carrier gas: N<sub>2</sub>.
- Injector temperature: 320°C.
- Detector temperature: 320°C.
- Column flow: 1 mL/min.
- Split ratio: 1:25.



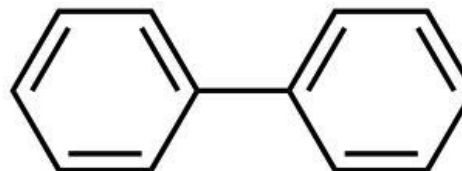
# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

Elution order:

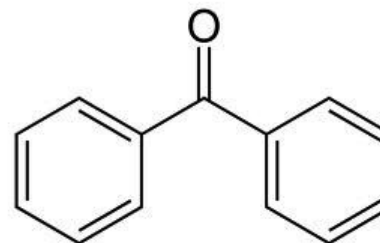
- Naphthalene (b.p. 218°C).



- Biphenyl (b.p. 255°C).



- Benzophenone (b.p. 305°C).



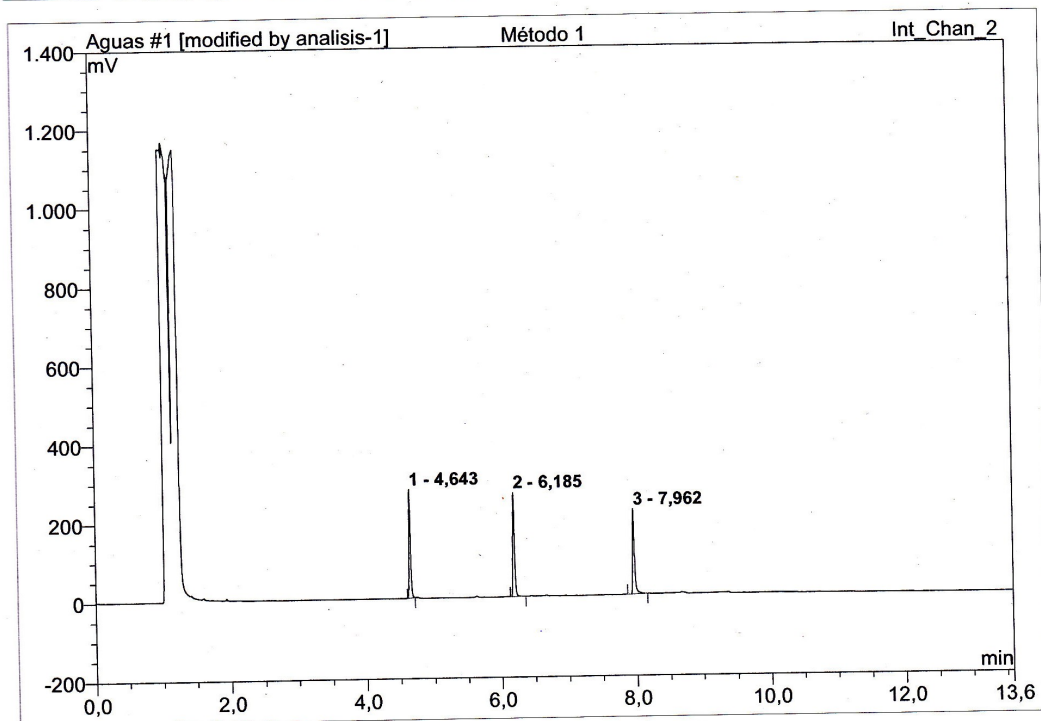
# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

## Method 1:

- Initial isotherm of 80°C during 1 min, then 20°C/min slope until 250°C and keep this temperature during 5 min. Total time: 14.5 min.

**1 Método 1**

|                  |                 |                   |             |
|------------------|-----------------|-------------------|-------------|
| Sample Name:     | Método 1        | Injection Volume: | 1,0         |
| Vial Number:     | 1               | Channel:          | Int_Chann_2 |
| Sample Type:     | unknown         | Wavelength:       | n.a.        |
| Control Program: | Aguas naftaleno | Bandwidth:        | n.a.        |
| Quantif. Method: | Aguas naftaleno | Dilution Factor:  | 1,0000      |
| Recording Time:  | 23/5/2011 21:01 | Sample Weight:    | 1,0000      |
| Run Time (min):  | 13,59           | Sample Amount:    | 1,0000      |



| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 4,64            | n.a.      | 276,852      | 7,637          | 34,51         | n.a.   | BMB* |
| 2      | 6,19            | n.a.      | 265,126      | 7,501          | 33,89         | n.a.   | BMB* |
| 3      | 7,96            | n.a.      | 217,275      | 6,995          | 31,61         | n.a.   | BMB* |
| Total: |                 |           | 759,253      | 22,133         | 100,00        | 0,000  |      |



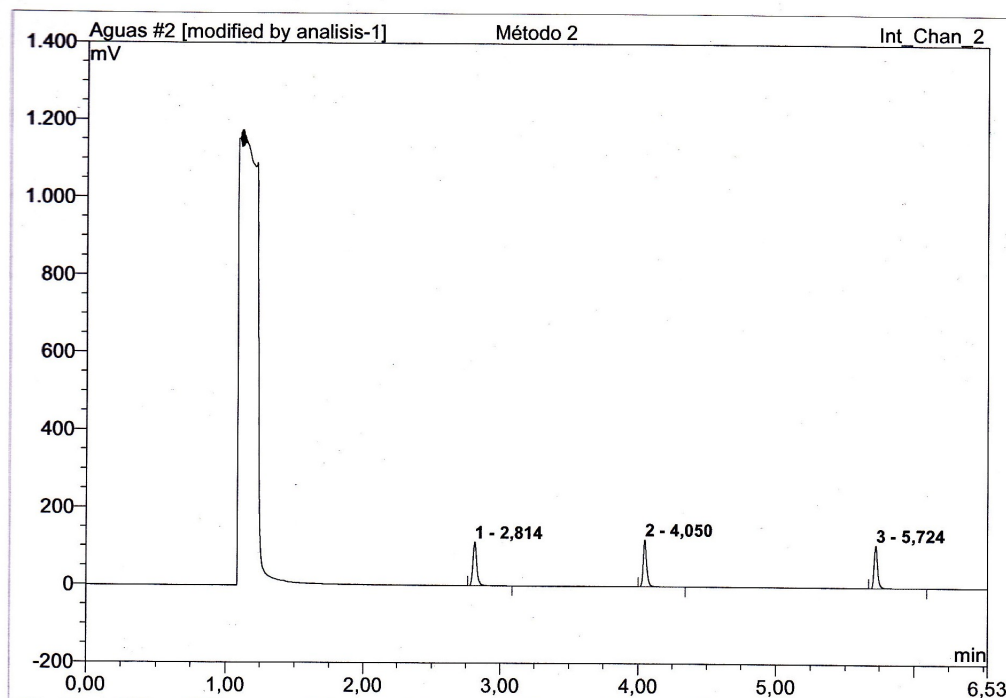
# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

## Method 2:

- Initial isotherm of 125°C during 1 min, then 20°C/min slope until 250°C and keep this temperature during 5 min. Total time: 12.25 min.

## 2 Método 2

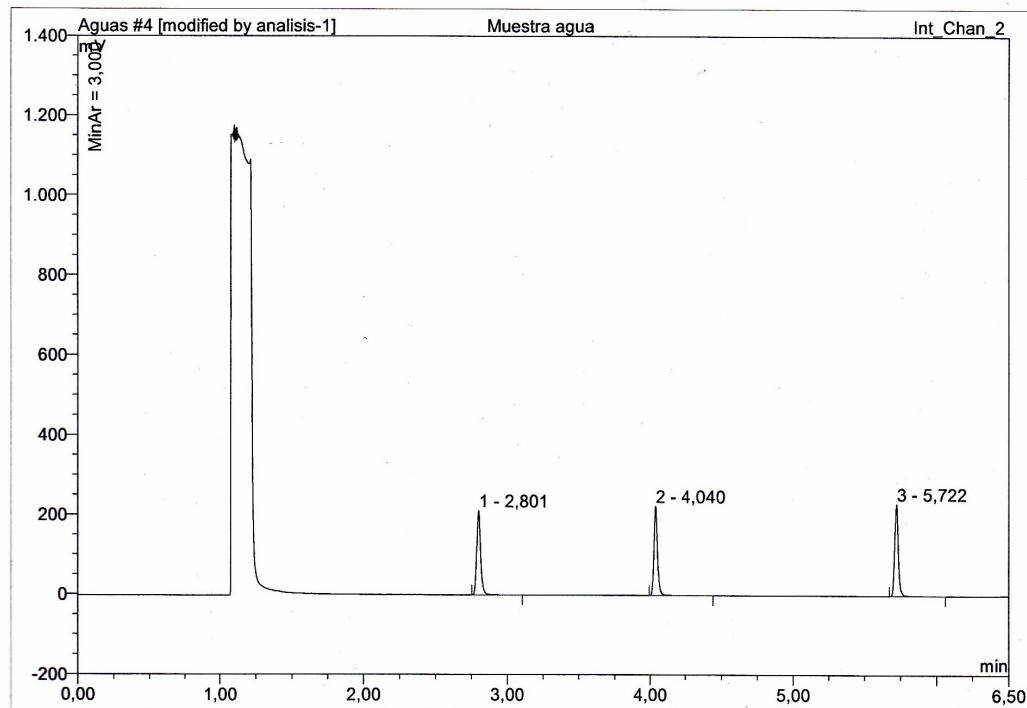
|                  |                 |                   |             |
|------------------|-----------------|-------------------|-------------|
| Sample Name:     | Método 2        | Injection Volume: | 1,0         |
| Vial Number:     | 2               | Channel:          | Int_Chann_2 |
| Sample Type:     | unknown         | Wavelength:       | n.a.        |
| Control Program: | Aguas naftaleno | Bandwidth:        | n.a.        |
| Quantif. Method: | Aguas naftaleno | Dilution Factor:  | 1,0000      |
| Recording Time:  | 23/5/2011 21:24 | Sample Weight:    | 1,0000      |
| Run Time (min):  | 6,53            | Sample Amount:    | 1,0000      |



| No.    | Ret. Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel. Area<br>% | Amount | Type |
|--------|------------------|-----------|--------------|----------------|----------------|--------|------|
| 1      | 2,81             | n.a.      | 112,536      | 3,722          | 35,03          | n.a.   | BMB  |
| 2      | 4,05             | n.a.      | 121,175      | 3,607          | 33,95          | n.a.   | BMB  |
| 3      | 5,72             | n.a.      | 110,253      | 3,296          | 31,02          | n.a.   | BMB  |
| Total: |                  |           | 343,963      | 10,625         | 100,00         | 0,000  |      |

#### 4 Muestra agua

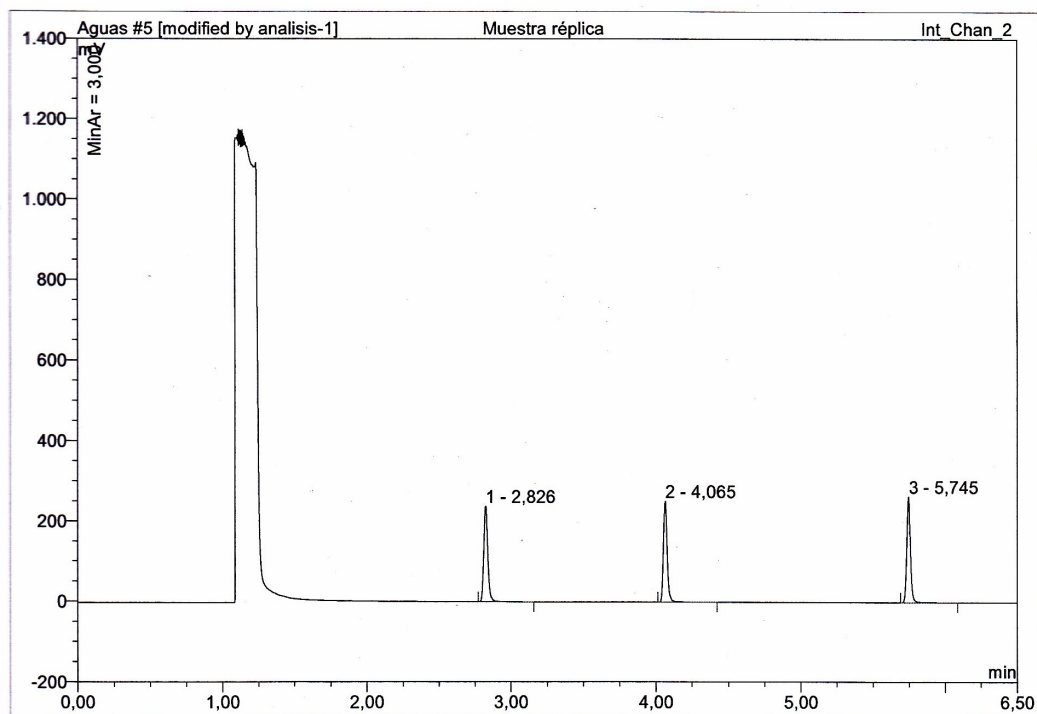
|                  |                 |                   |                |
|------------------|-----------------|-------------------|----------------|
| Sample Name:     | Muestra agua    | Injection Volume: | 1,0            |
| Vial Number:     | 4               | Channel:          | Int_Channels_2 |
| Sample Type:     | unknown         | Wavelength:       | n.a.           |
| Control Program: | Aguas naftaleno | Bandwidth:        | n.a.           |
| Quantif. Method: | Aguas naftaleno | Dilution Factor:  | 1,0000         |
| Recording Time:  | 24/5/2011 18:07 | Sample Weight:    | 1,0000         |
| Run Time (min):  | 6,50            | Sample Amount:    | 1,0000         |



| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,80            | n.a.      | 211,430      | 6,756          | 33,87         | n.a.   | BMB  |
| 2      | 4,04            | n.a.      | 224,478      | 6,567          | 32,92         | n.a.   | BMB  |
| 3      | 5,72            | n.a.      | 230,969      | 6,624          | 33,21         | n.a.   | BMB  |
| Total: |                 |           | 666,876      | 19,947         | 100,00        | 0,000  |      |

## 5 Muestra réplica

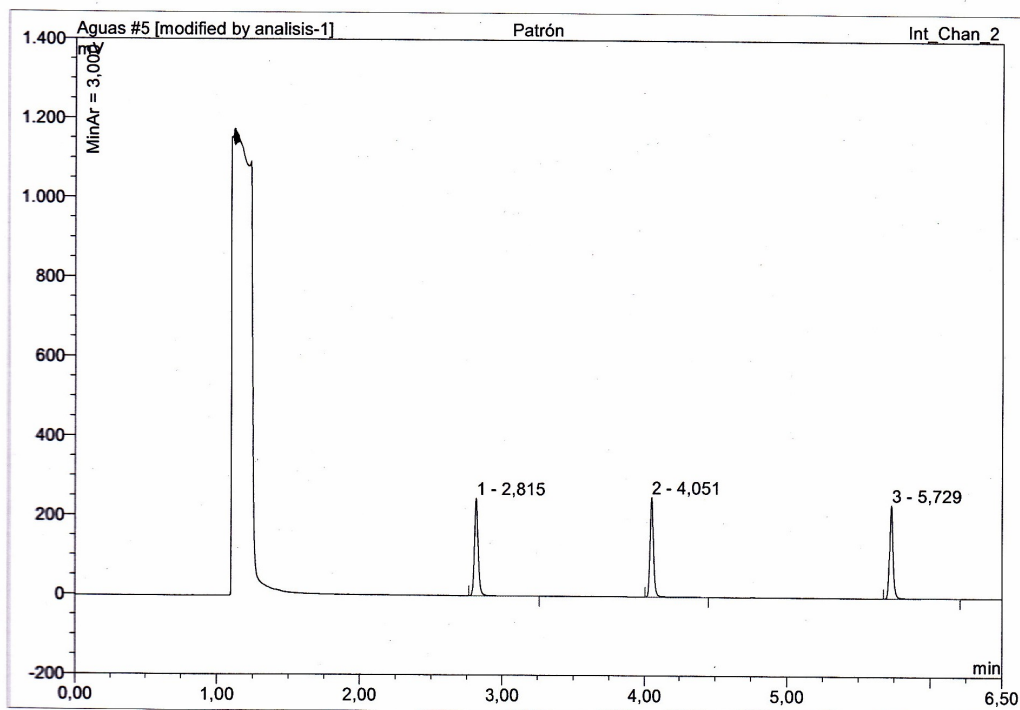
|                  |                 |                   |                |
|------------------|-----------------|-------------------|----------------|
| Sample Name:     | Muestra réplica | Injection Volume: | 1,0            |
| Vial Number:     | 6               | Channel:          | Int_Channels_2 |
| Sample Type:     | unknown         | Wavelength:       | n.a.           |
| Control Program: | Aguas naftaleno | Bandwidth:        | n.a.           |
| Quantif. Method: | Aguas naftaleno | Dilution Factor:  | 1,0000         |
| Recording Time:  | 24/5/2011 19:27 | Sample Weight:    | 1,0000         |
| Run Time (min):  | 6,50            | Sample Amount:    | 1,0000         |



| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,83            | n.a.      | 238,344      | 7,729          | 33,98         | n.a.   | BMB  |
| 2      | 4,07            | n.a.      | 251,342      | 7,463          | 32,81         | n.a.   | BMB  |
| 3      | 5,75            | n.a.      | 263,888      | 7,557          | 33,22         | n.a.   | BMB  |
| Total: |                 |           | 753,574      | 22,750         | 100,00        | 0,000  |      |

## 5 Patrón

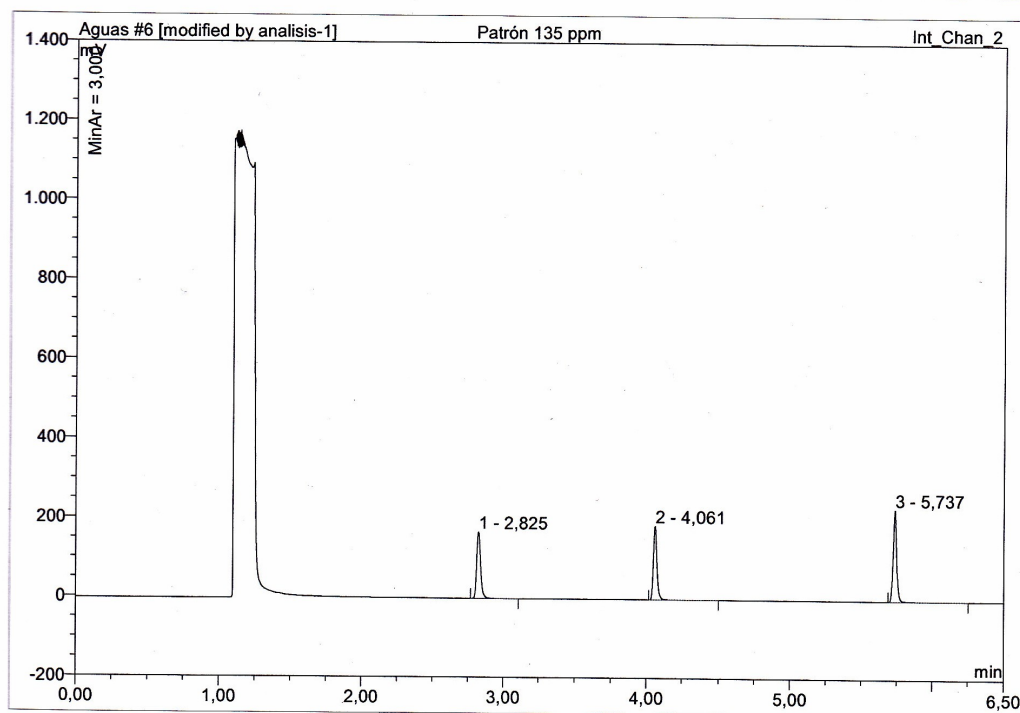
|                  |                 |                   |             |
|------------------|-----------------|-------------------|-------------|
| Sample Name:     | Patrón          | Injection Volume: | 1,0         |
| Vial Number:     | 5               | Channel:          | Int_Chann_2 |
| Sample Type:     | unknown         | Wavelength:       | n.a.        |
| Control Program: | Aguas naftaleno | Bandwidth:        | n.a.        |
| Quantif. Method: | Aguas naftaleno | Dilution Factor:  | 1,0000      |
| Recording Time:  | 24/5/2011 18:23 | Sample Weight:    | 1,0000      |
| Run Time (min):  | 6,50            | Sample Amount:    | 1,0000      |



| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,81            | n.a.      | 246,380      | 7,408          | 34,87         | n.a.   | BMB  |
| 2      | 4,05            | n.a.      | 252,091      | 7,229          | 34,03         | n.a.   | BMB  |
| 3      | 5,73            | n.a.      | 235,423      | 6,609          | 31,11         | n.a.   | BMB  |
| Total: |                 |           | 733,894      | 21,246         | 100,00        | 0,000  |      |

**6 Patrón 135 ppm**

|                         |                        |                          |                    |
|-------------------------|------------------------|--------------------------|--------------------|
| <b>Sample Name:</b>     | <b>Patrón 135 ppm</b>  | <b>Injection Volume:</b> | <b>1,0</b>         |
| <b>Vial Number:</b>     | <b>7</b>               | <b>Channel:</b>          | <b>Int_Chann_2</b> |
| <b>Sample Type:</b>     | <b>unknown</b>         | <b>Wavelength:</b>       | <b>n.a.</b>        |
| <b>Control Program:</b> | <b>Aguas naftaleno</b> | <b>Bandwidth:</b>        | <b>n.a.</b>        |
| <b>Quantif. Method:</b> | <b>Aguas naftaleno</b> | <b>Dilution Factor:</b>  | <b>1,0000</b>      |
| <b>Recording Time:</b>  | <b>24/5/2011 20:41</b> | <b>Sample Weight:</b>    | <b>1,0000</b>      |
| <b>Run Time (min):</b>  | <b>6,50</b>            | <b>Sample Amount:</b>    | <b>1,0000</b>      |

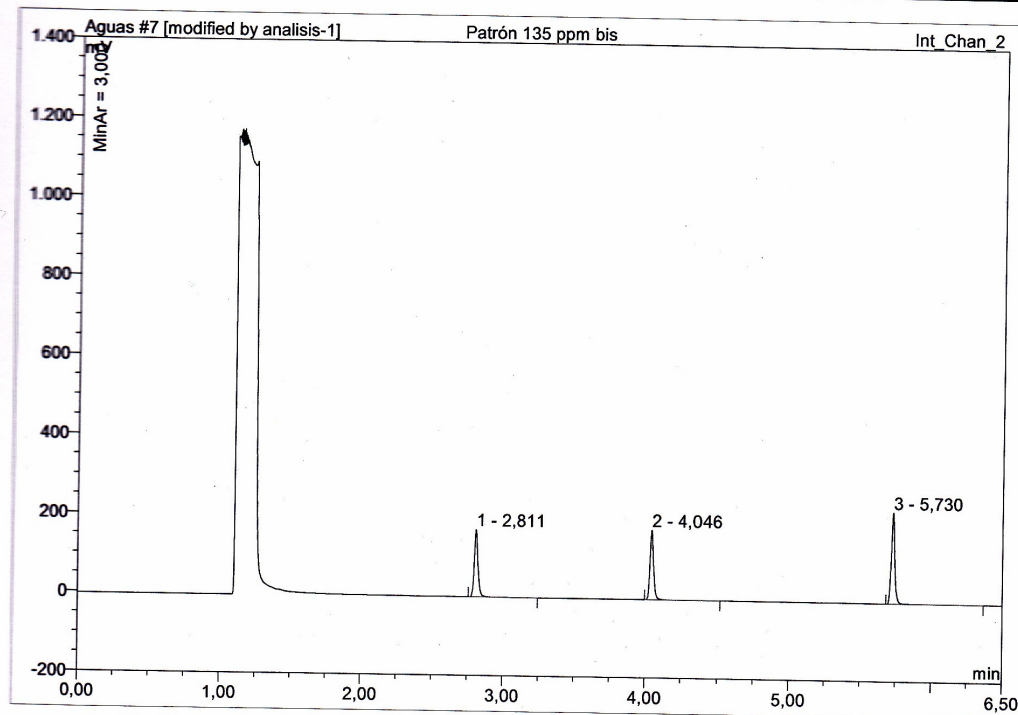


| No.           | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|---------------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1             | 2,82            | n.a.      | 167,532      | 5,288          | 30,73         | n.a.   | BMB  |
| 2             | 4,06            | n.a.      | 185,581      | 5,250          | 30,51         | n.a.   | BMB  |
| 3             | 5,74            | n.a.      | 231,031      | 6,670          | 38,76         | n.a.   | BMB  |
| <b>Total:</b> |                 |           | 584,144      | 17,209         | 100,00        | 0,000  |      |



**7 Patrón 135 ppm bis**

|                  |                    |                   |              |
|------------------|--------------------|-------------------|--------------|
| Sample Name:     | Patrón 135 ppm bis | Injection Volume: | 1,0          |
| Vial Number:     | 8                  | Channel:          | Int_Chanel_2 |
| Sample Type:     | unknown            | Wavelength:       | n.a.         |
| Control Program: | Aguas naftaleno    | Bandwidth:        | n.a.         |
| Quantif. Method: | Aguas naftaleno    | Dilution Factor:  | 1,0000       |
| Recording Time:  | 24/5/2011 20:53    | Sample Weight:    | 1,0000       |
| Run Time (min):  | 6,50               | Sample Amount:    | 1,0000       |

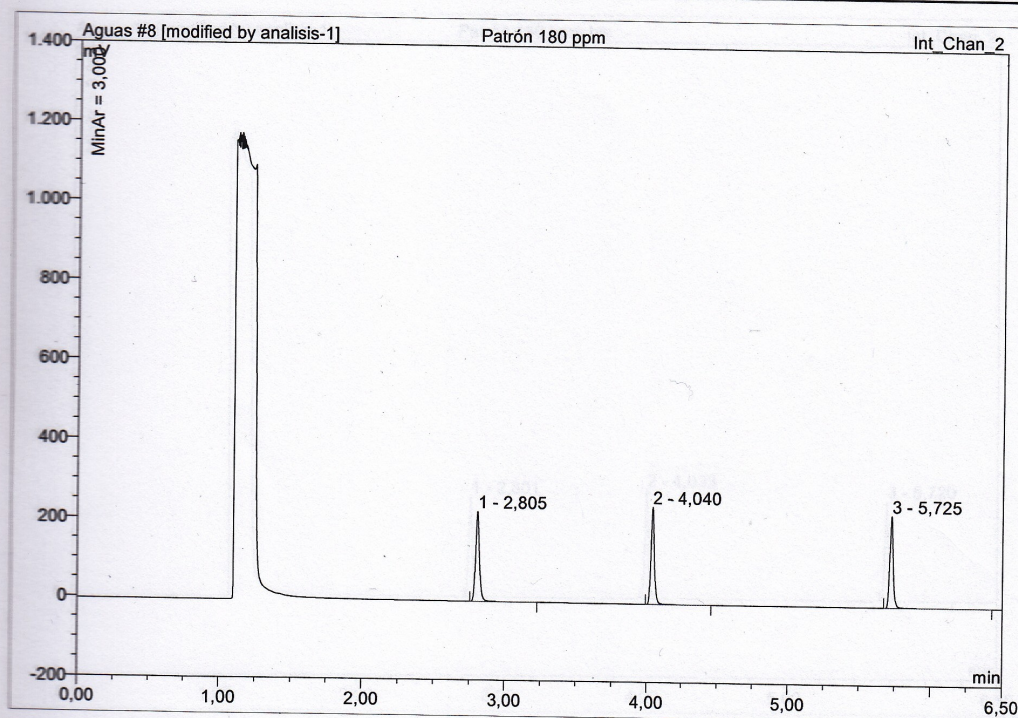


| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,81            | n.a.      | 170,692      | 5,285          | 30,90         | n.a.   | BMB  |
| 2      | 4,05            | n.a.      | 176,506      | 5,205          | 30,43         | n.a.   | BMB  |
| 3      | 5,73            | n.a.      | 231,333      | 6,614          | 38,67         | n.a.   | BMB  |
| Total: |                 |           | 578,531      | 17,105         | 100,00        | 0,000  |      |

**8 Patrón 180 ppm**

Sample Name: Patrón 180 ppm  
Vial Number: 9  
Sample Type: unknown  
Control Program: Aguas naftaleno  
Quantif. Method: Aguas naftaleno  
Recording Time: 24/5/2011 21:04  
Run Time (min): 6,50

Injection Volume: 1,0  
Channel: Int\_Chann\_2  
Wavelength: n.a.  
Bandwidth: n.a.  
Dilution Factor: 1,0000  
Sample Weight: 1,0000  
Sample Amount: 1,0000

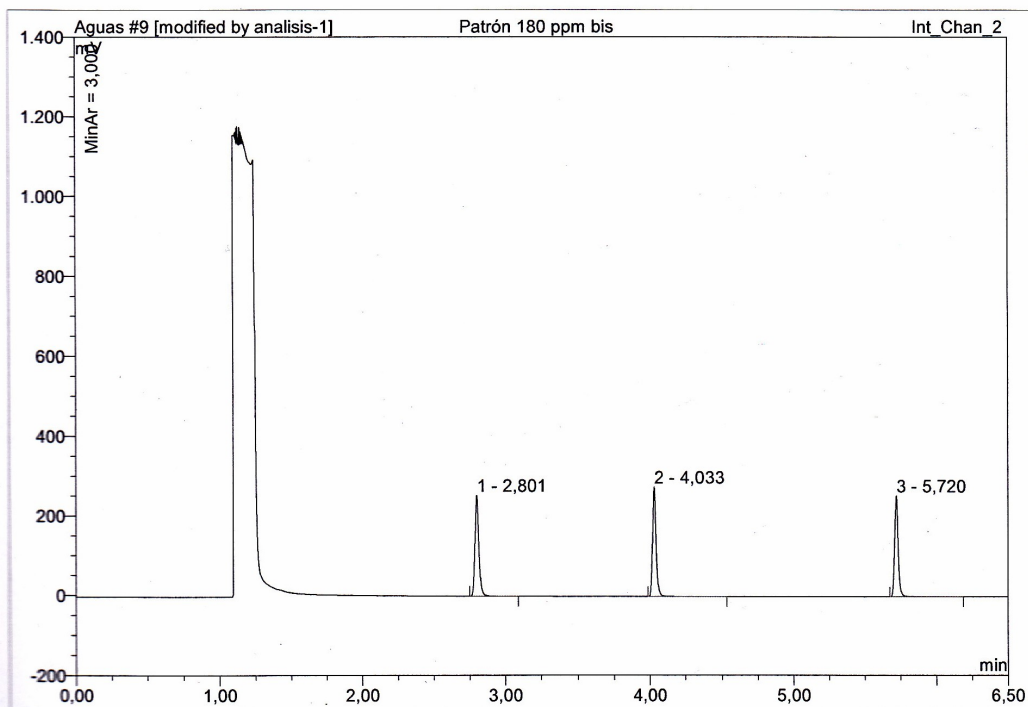


| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,81            | n.a.      | 227,575      | 7,184          | 34,34         | n.a.   | BMB  |
| 2      | 4,04            | n.a.      | 245,310      | 7,114          | 34,01         | n.a.   | BMB  |
| 3      | 5,73            | n.a.      | 231,784      | 6,619          | 31,64         | n.a.   | BMB  |
| Total: |                 |           | 704,669      | 20,917         | 100,00        | 0,000  |      |



**9 Patrón 180 ppm bis**

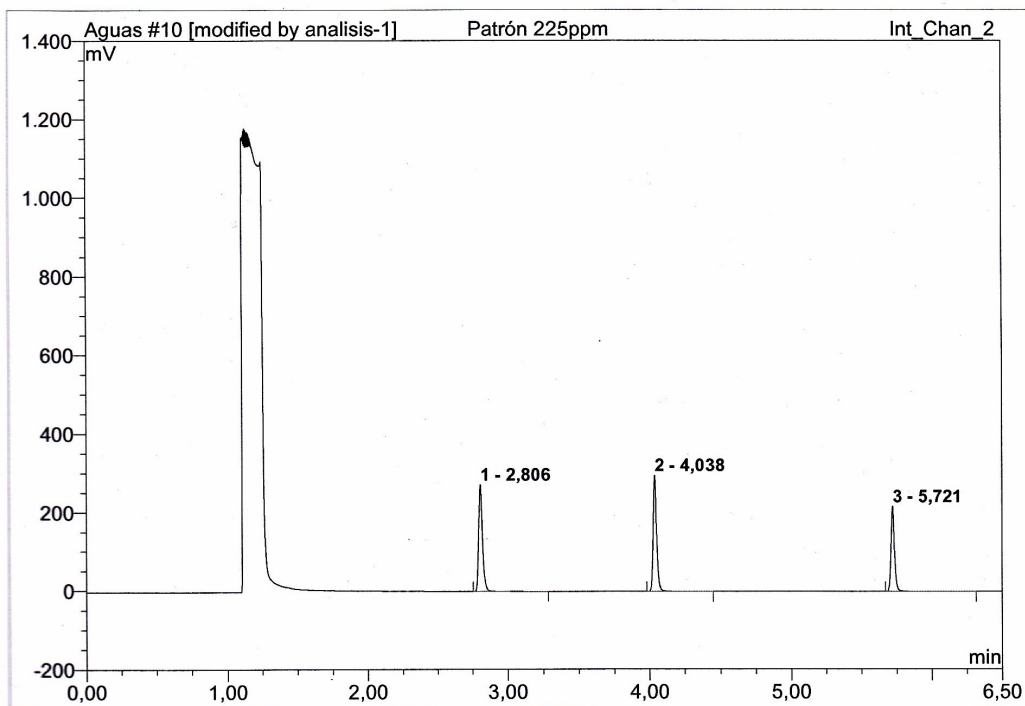
|                  |                    |                   |             |
|------------------|--------------------|-------------------|-------------|
| Sample Name:     | Patrón 180 ppm bis | Injection Volume: | 1,0         |
| Vial Number:     | 10                 | Channel:          | Int_Chann_2 |
| Sample Type:     | unknown            | Wavelength:       | n.a.        |
| Control Program: | Aguas naftaleno    | Bandwidth:        | n.a.        |
| Quantif. Method: | Aguas naftaleno    | Dilution Factor:  | 1,0000      |
| Recording Time:  | 24/5/2011 21:14    | Sample Weight:    | 1,0000      |
| Run Time (min):  | 6,50               | Sample Amount:    | 1,0000      |



| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,80            | n.a.      | 253,148      | 7,957          | 34,44         | n.a.   | BMB  |
| 2      | 4,03            | n.a.      | 274,857      | 7,868          | 34,06         | n.a.   | BMB  |
| 3      | 5,72            | n.a.      | 253,397      | 7,279          | 31,51         | n.a.   | BMB  |
| Total: |                 |           | 781,402      | 23,105         | 100,00        | 0,000  |      |

**10 Patrón 225ppm**

|                  |                 |                   |          |
|------------------|-----------------|-------------------|----------|
| Sample Name:     | Patrón 225ppm   | Injection Volume: | 1,0      |
| Vial Number:     | 11              | Channel:          | Int_Ch_2 |
| Sample Type:     | unknown         | Wavelength:       | n.a.     |
| Control Program: | Aguas naftaleno | Bandwidth:        | n.a.     |
| Quantif. Method: | Aguas naftaleno | Dilution Factor:  | 1,0000   |
| Recording Time:  | 24/5/2011 21:24 | Sample Weight:    | 1,0000   |
| Run Time (min):  | 6,50            | Sample Amount:    | 1,0000   |

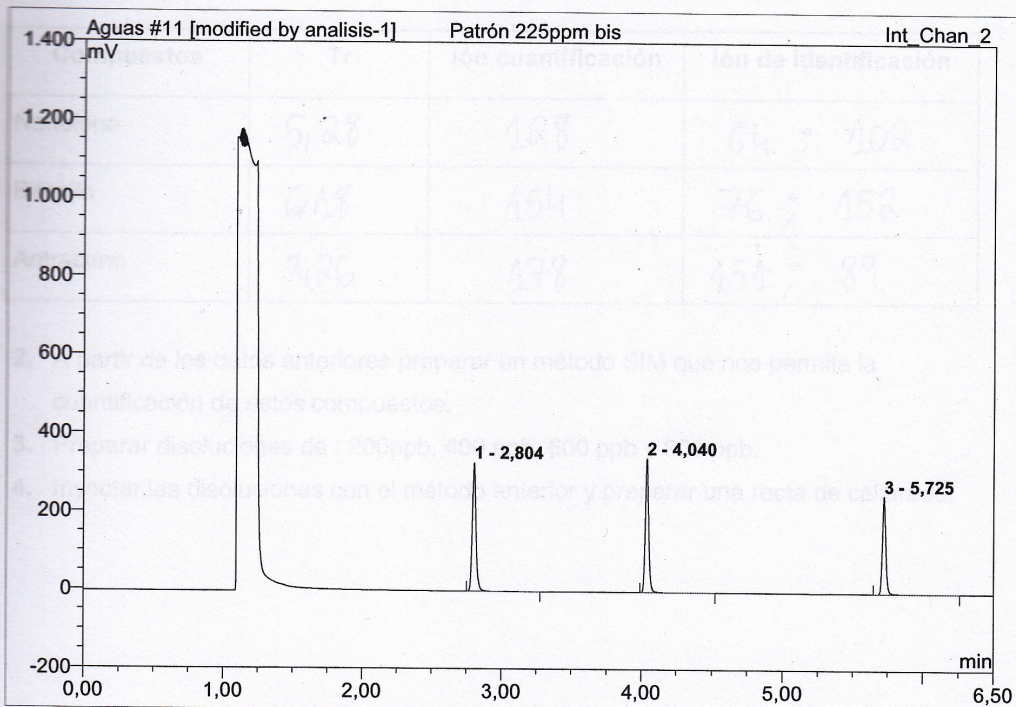


| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,81            | n.a.      | 272,348      | 8,561          | 36,65         | n.a.   | BMB  |
| 2      | 4,04            | n.a.      | 296,461      | 8,550          | 36,60         | n.a.   | BMB  |
| 3      | 5,72            | n.a.      | 217,809      | 6,250          | 26,75         | n.a.   | BMB  |
| Total: |                 |           | 786,617      | 23,360         | 100,00        | 0,000  |      |

**11 Patrón 225ppm bis**

Preparación de un método SIM para el análisis de PAHs.

|                  |                   |                   |             |
|------------------|-------------------|-------------------|-------------|
| Sample Name:     | Patrón 225ppm bis | Injection Volume: | 1,0         |
| Vial Number:     | 12                | Channel:          | Int_Chann_2 |
| Sample Type:     | unknown           | Wavelength:       | n.a.        |
| Control Program: | Aguas naftaleno   | Bandwidth:        | n.a.        |
| Quantif. Method: | Aguas naftaleno   | Dilution Factor:  | 1,0000      |
| Recording Time:  | 24/5/2011 21:35   | Sample Weight:    | 1,0000      |
| Run Time (min):  | 6,50              | Sample Amount:    | 1,0000      |



| No.    | Ret.Time<br>min | Peak Name | Height<br>mV | Area<br>mV*min | Rel.Area<br>% | Amount | Type |
|--------|-----------------|-----------|--------------|----------------|---------------|--------|------|
| 1      | 2,80            | n.a.      | 328,938      | 9,934          | 36,95         | n.a.   | BMB  |
| 2      | 4,04            | n.a.      | 342,561      | 9,805          | 36,47         | n.a.   | BMB  |
| 3      | 5,73            | n.a.      | 250,600      | 7,145          | 26,58         | n.a.   | BMB  |
| Total: |                 |           | 922,099      | 26,883         | 100,00        | 0,000  |      |

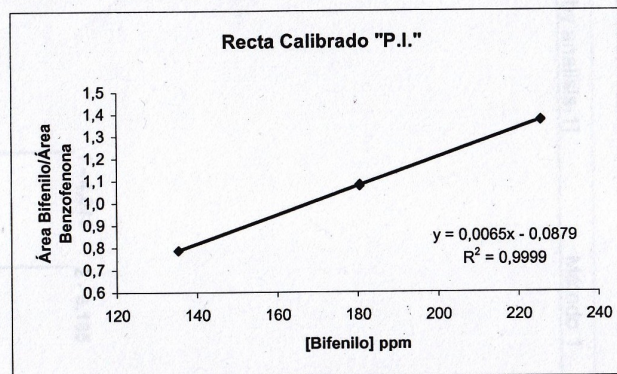
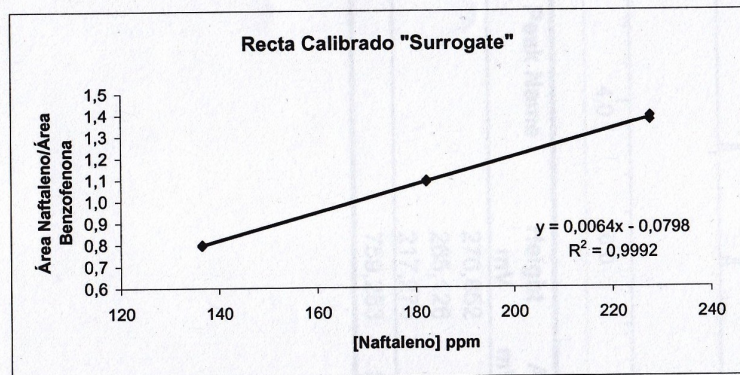


# ANALYSIS OF PAHs IN WATER BY SPE-GC-FID

## Results:

### DETERMINACIÓN DE NAFTALENO EN H<sub>2</sub>O

| Concentración Naftaleno (ppm) | Concentración Bifenilo (ppm) | Concentración Benzofenona (ppm) | Área Naftaleno | Área Bifenilo | Área Benzofenona | Área Naftaleno/Área Benzofenona | Área Bifenilo/Área Benzofenona |
|-------------------------------|------------------------------|---------------------------------|----------------|---------------|------------------|---------------------------------|--------------------------------|
| 136,56                        | 135,36                       | 200,80                          | 5,29           | 5,25          | 6,67             | 0,79                            | 0,79                           |
| 136,56                        | 135,36                       | 200,80                          | 5,29           | 5,21          | 6,61             | 0,80                            | 0,79                           |
| 182,08                        | 180,48                       | 200,80                          | 7,18           | 7,11          | 6,62             | 1,09                            | 1,07                           |
| 182,08                        | 180,48                       | 200,80                          | 7,96           | 7,87          | 7,28             | 1,09                            | 1,08                           |
| 227,60                        | 225,60                       | 200,80                          | 8,56           | 8,55          | 6,25             | 1,37                            | 1,37                           |
| 227,60                        | 225,60                       | 200,80                          | 9,93           | 9,81          | 7,15             | 1,39                            | 1,37                           |



| Área Bifenilo en la muestra | Área Benzofenona en la muestra | Relación de áreas | Concentración de Bifenilo en la muestra (ppm) | Recuperación % |
|-----------------------------|--------------------------------|-------------------|-----------------------------------------------|----------------|
| 6,57                        | 6,62                           | 0,99              | 167,37                                        | 83             |
| 7,46                        | 7,56                           | 0,99              | 166,78                                        | 83             |

| Área Naftaleno en la muestra | Área Benzofenona en la muestra | Relación de áreas | Concentración de Naftaleno en la muestra (ppm) | Naftaleno en Agua (ppm) |
|------------------------------|--------------------------------|-------------------|------------------------------------------------|-------------------------|
| 6,76                         | 6,62                           | 1,02              | 170,44                                         | 5                       |
| 7,73                         | 7,56                           | 1,02              | 170,87                                         | 4,8                     |

# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

Aim: Determining naphthalene, biphenyl and anthracene in water samples (present in around 500 ppb each one).

Steps:

- Solution 500 ppm of each one in isooctane.
- Injection in splitless mode (splitless time: 2 min).
- Develop a chromatographic method for solving them.
- Select the most important ions of the spectrum of each compound.
- Prepare solutions in concentrations: 200 ppb, 400 ppb, 600 ppb and 800 ppb.
- Identification and quantitation of the analytes by SIM.

# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

## Equipment:

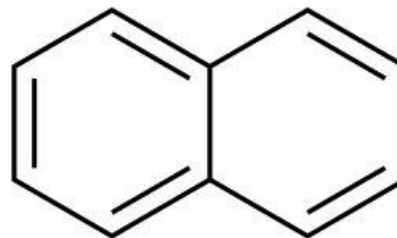
- Gas Chromatograph GC 17-A Shimadzu (Sh 17).
- Column: TBR-5 (30 m x 0.25 id x 0.25 $\mu$ m sp).
- Stationary phase: DB-5 (5% phenyl–95%methylsyloxane).
- Carrier gas: He.
- Injector temperature: 320°C.
- Detector temperature: 320°C.
- Column flow: 1 mL/min.
- Splitless time: 2 min.



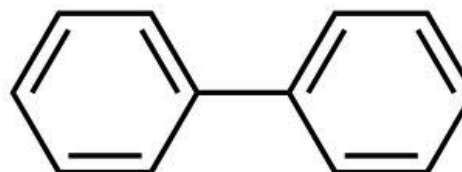
# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

Elution order:

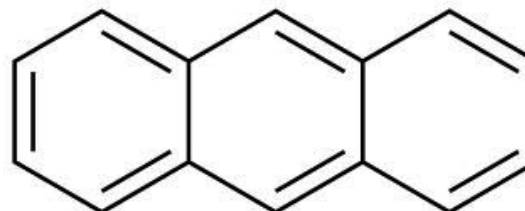
- Naphthalene (b.p. 218°C).



- Biphenyl (b.p. 255°C).



- Anthracene (b.p. 340°C).





# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

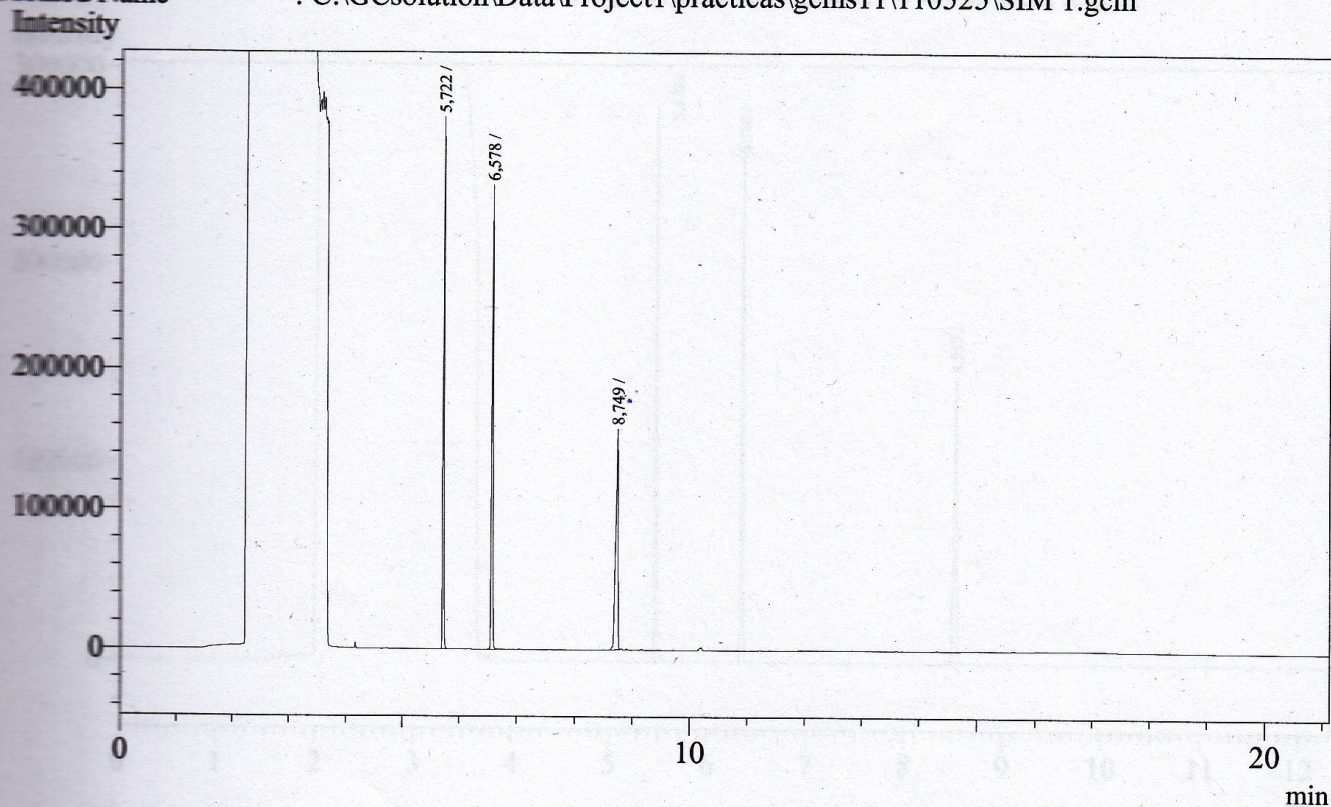
## Method 1:

- Initial isotherm of 50°C during 2 min, then 40°C/min slope until 20°C and another slope of 15°C/min until 350°C, then keep this temperature during 5 min. Total time: 20 min.

Analysis Date & Time : 25/05/2011 19:02:36  
User Name : Admin  
Vial# : 1  
Sample Name : Método SIM  
Sample ID :  
Sample Type : Unknown  
Injection Volume :  
ISTD Amount :

MÉTODO 1

Data Name : C:\GCsolution\Data\Project1\praticas\gcms11\110525\SIM 1.gcd  
Method Name : C:\GCsolution\Data\Project1\praticas\gcms11\110525\SIM 1.gcm



| Peak# | Ret.Time | Area    | Height | Conc.  | Unit | Mark | ID# | Cmpd Name |
|-------|----------|---------|--------|--------|------|------|-----|-----------|
| 1     | 5,722    | 485116  | 371234 | 33,363 |      |      |     |           |
| 2     | 6,578    | 531674  | 324493 | 36,565 |      |      |     |           |
| 3     | 8,749    | 437259  | 147609 | 30,072 |      |      |     |           |
| Total |          | 1454049 | 843336 |        |      |      |     |           |

# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

## Method 2:

- Initial isotherm of 60°C during 2 min, then 40°C/min slope until 200°C and another slope of 15°C/min until 300°C, then keep this temperature during 5 min. Total time: 12 min.



Analysis Date & Time : 25/05/2011 19:45:10

User Name : Admin

Vial# : 1

Sample Name : Método SIM

Sample ID :

Sample Type : Unknown

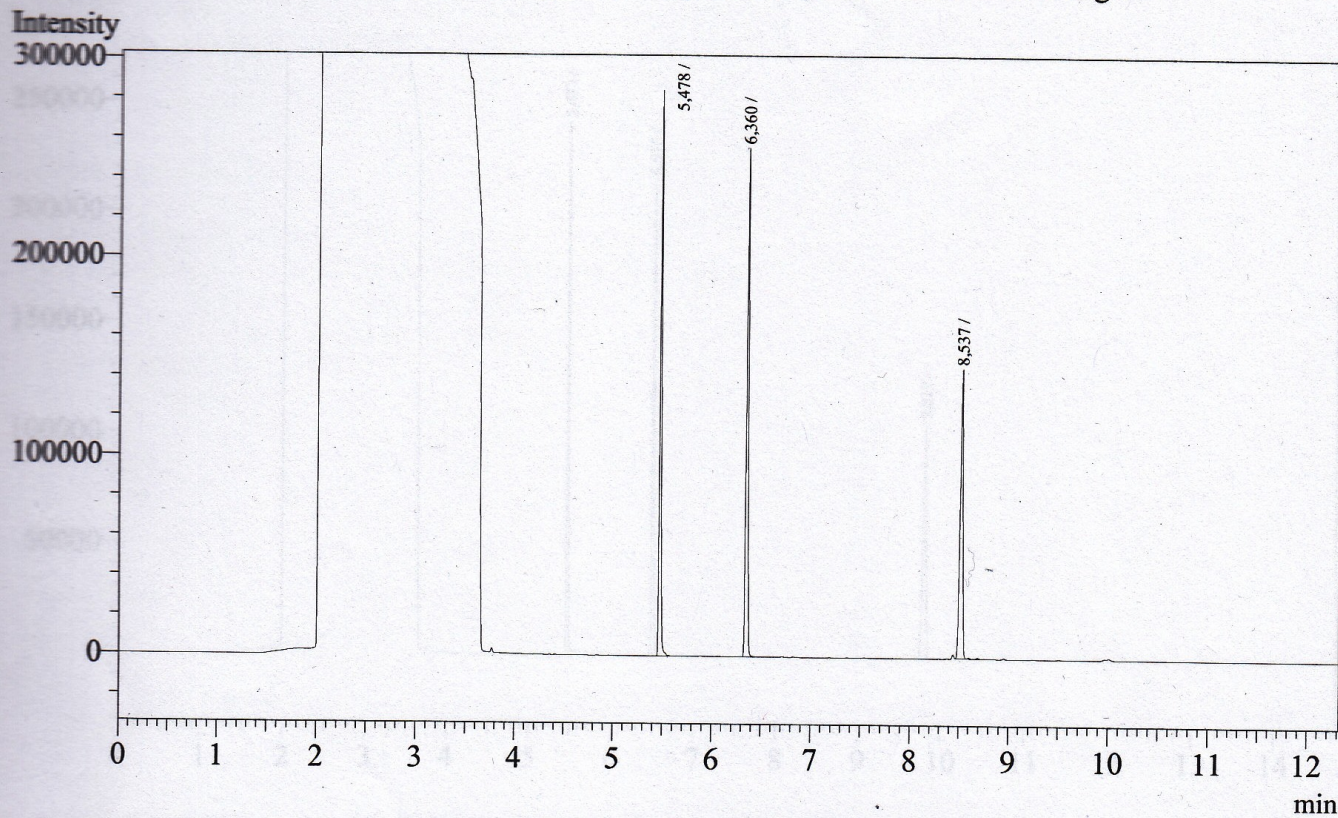
Injection Volume :

ISTD Amount :

MÉTODO 2

Data Name : C:\GCsolution\Data\Project1\praticas\gcms11\110525\SIM 2.gcd

Method Name : C:\GCsolution\Data\Project1\praticas\gcms11\110525\SIM 2.gcm



| Peak# | Ret.Time | Area   | Height | Conc.  | Unit | Mark | ID# | Cmpd Name |
|-------|----------|--------|--------|--------|------|------|-----|-----------|
| 1     | 5.478    | 325024 | 279039 | 33.449 |      |      |     |           |
| 2     | 6.360    | 357278 | 245025 | 36.769 |      |      |     |           |
| 3     | 8.537    | 289393 | 143678 | 29.782 |      | S    |     |           |
| Total |          | 971695 | 667742 |        |      |      |     |           |

# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

## Method 3:

- Initial isotherm of 60°C during 2 min, then 40°C/min slope until 190°C, isotherm at this temperature during 2 min and another slope of 15°C/min until 300°C. Total time: 12 min.



Analysis Date & Time : 25/05/2011 20:06:51

User Name : Admin

Vial# : 1

Sample Name : Método SIM

Sample ID :

Sample Type : Unknown

Injection Volume :

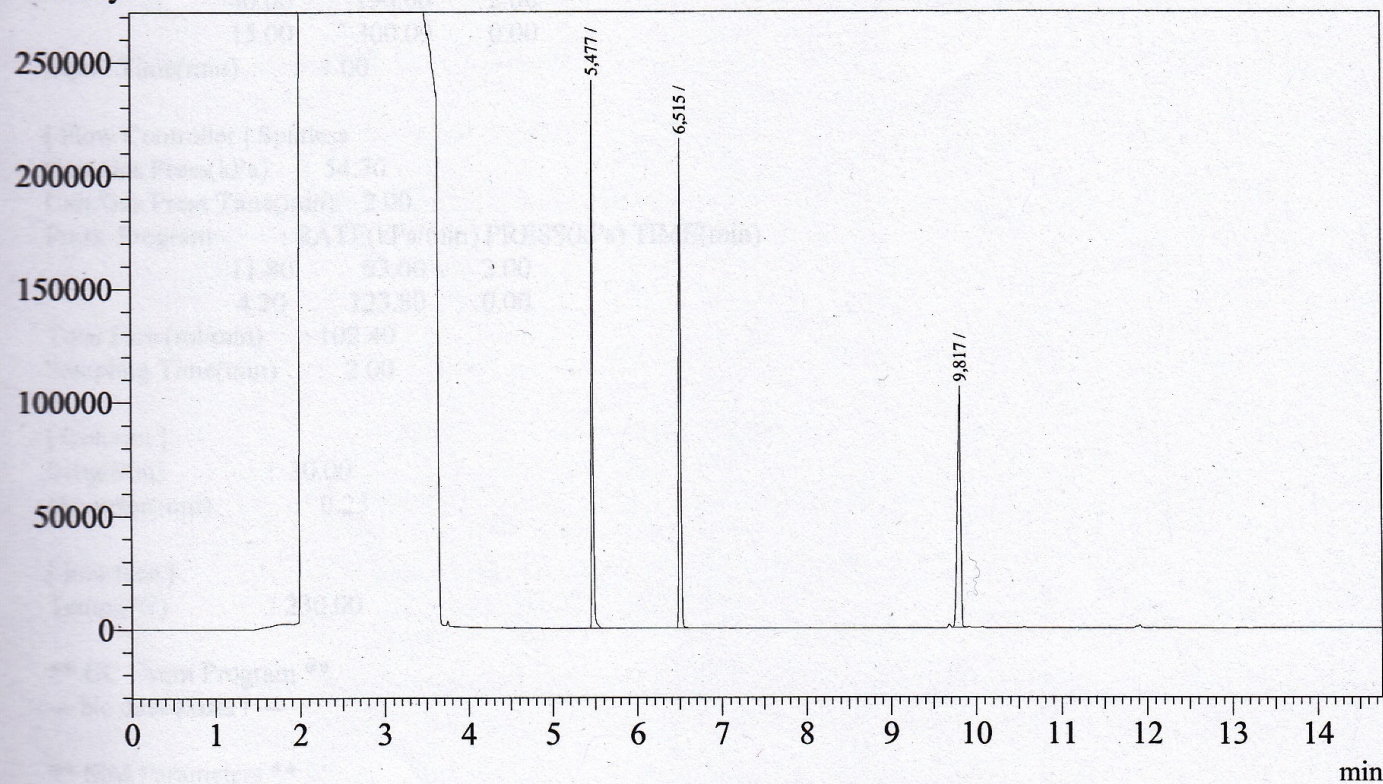
ISTD Amount :

MÉTODO 3

Data Name : C:\GCsolution\Data\Project1\practicass\gcms11\110525\SIM 4.gcd

Method Name : C:\GCsolution\Data\Project1\practicass\gcms11\110525\SIM 3.gcm

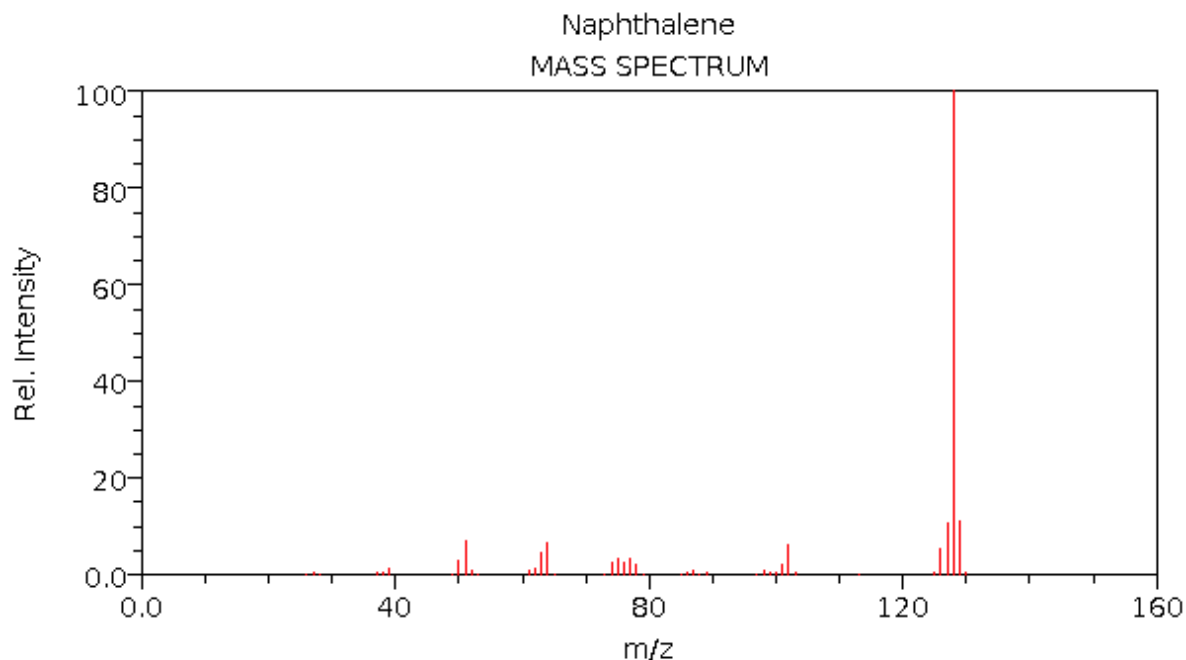
Intensity



| Peak# | Ret.Time | Area   | Height | Conc.  | Unit | Mark | ID# | Cmpd Name |
|-------|----------|--------|--------|--------|------|------|-----|-----------|
| 1     | 5,477    | 313525 | 236313 | 33,544 |      |      |     |           |
| 2     | 6,515    | 342114 | 205262 | 36,602 |      |      |     |           |
| 3     | 9,817    | 279036 | 105509 | 29,854 |      |      |     |           |
| Total |          | 934675 | 547084 |        |      |      |     |           |



# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

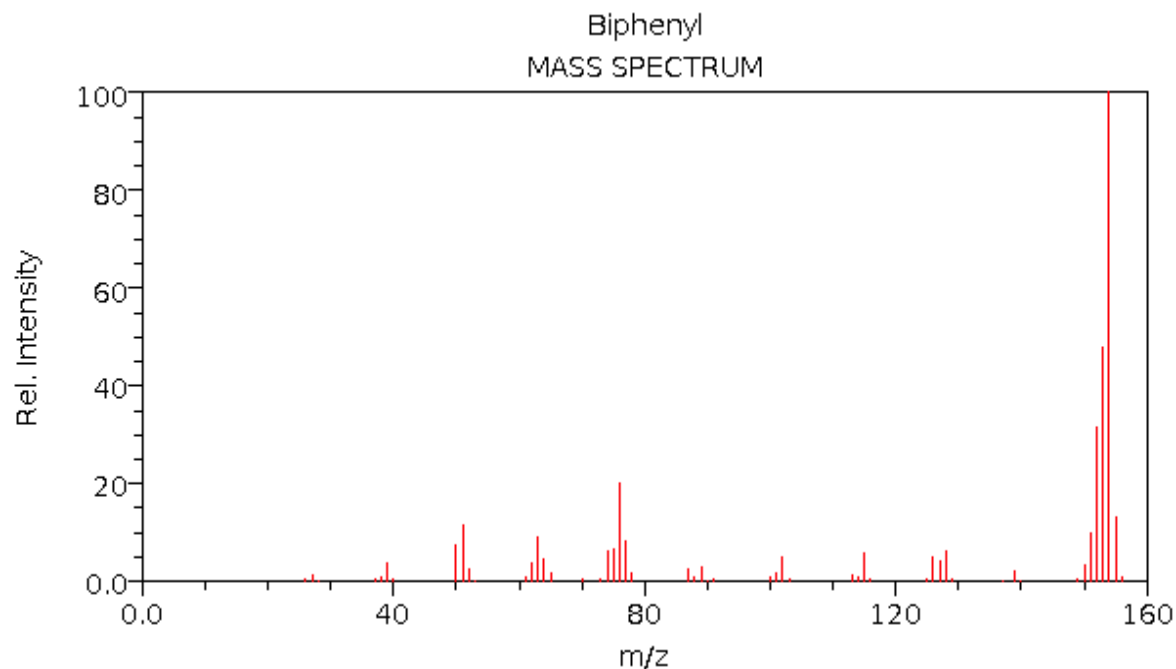


NIST Chemistry WebBook (<http://webbook.nist.gov/chemistry>)

## Ions taken:

- For quantitation: 128.
- For identification: 64, 102.

# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)

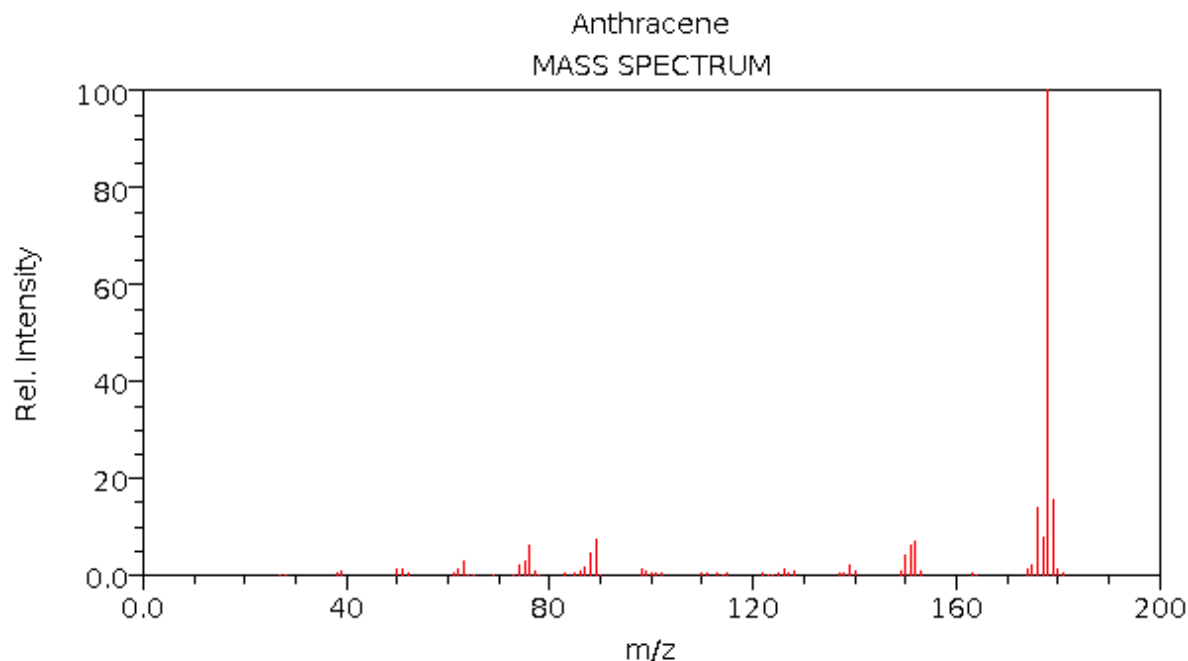


NIST Chemistry WebBook (<http://webbook.nist.gov/chemistry>)

Ions taken:

- For quantitation: 154.
- For identification: 76, 152.

# ANALYSIS OF PAHs IN WATER BY GC/MS (SIM MODE)



NIST Chemistry WebBook (<http://webbook.nist.gov/chemistry>)

Ions taken:

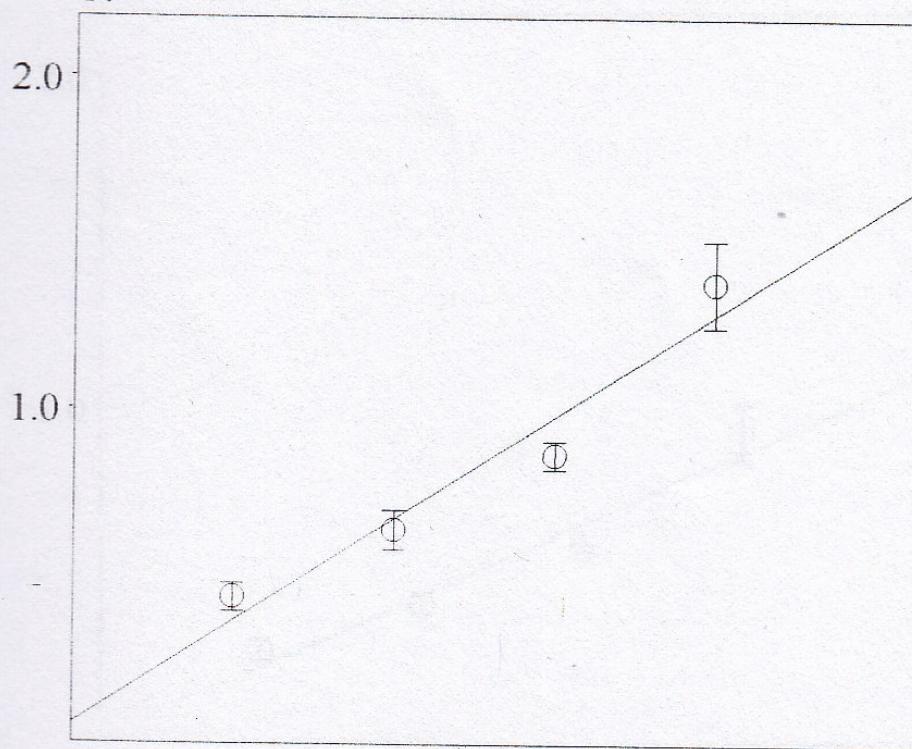
- For quantitation: 178.
- For identification: 89, 151.

ID # 1 M/Z : 128.00 Name : naftaleno

Area = 148.795 \* (Conc.) + 6215 r2 = 0.942528

Area

10<sup>5</sup>



0

0.5

1.0

Conc

10<sup>3</sup>

Conc.(ppb )

Area

|   |         |        |
|---|---------|--------|
| 1 | 206.400 | 43487  |
| 2 | 412.800 | 63790  |
| 3 | 619.200 | 86361  |
| 4 | 825.600 | 138334 |



ID # 2 M/Z : 154.00 Name : bifenilo

Area = 101.289 \* (Conc.) + 2702 r2 = 0.949897

Area

1(5

2.0

1.0

0

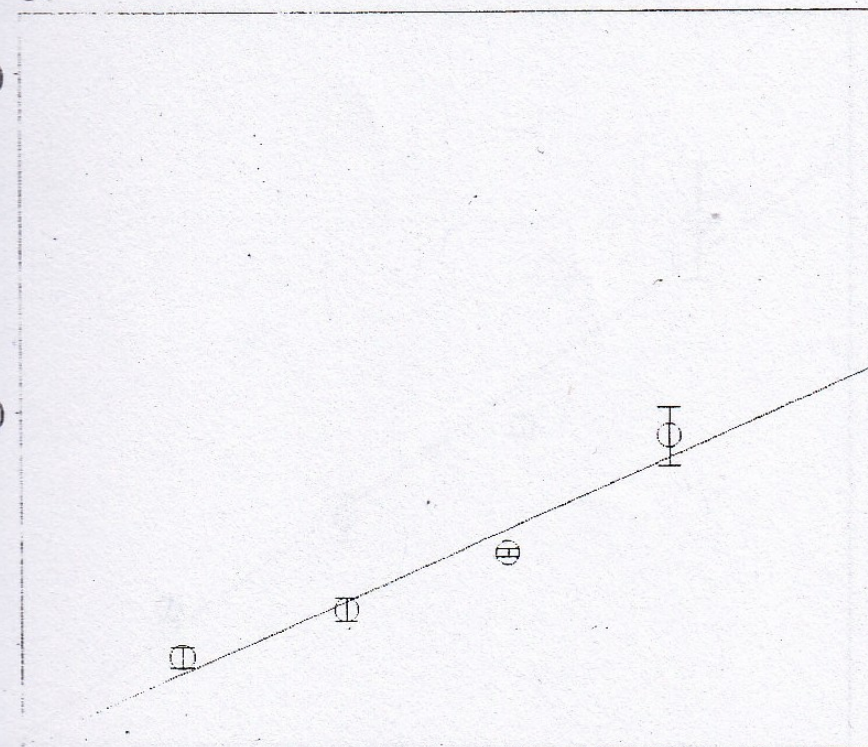
0.5

1.0

Conc

10<sup>3</sup>

|   | Conc.(ppb ) | Area  |
|---|-------------|-------|
| 1 | 206.400     | 27943 |
| 2 | 412.800     | 41696 |
| 3 | 619.200     | 58052 |
| 4 | 825.600     | 92178 |





ID # 3 M/Z : 178.00 Name : antraceno

Area = 83.4906 \* (Conc.) + -2107 r2 = 0.954230

Area

10<sup>5</sup>

1.0

0.5

0

0.5

Conc

1.0

10<sup>3</sup>

Conc.(ppb )

Area

|   |         |       |
|---|---------|-------|
| 1 | 224.000 | 19921 |
| 2 | 448.000 | 33661 |
| 3 | 672.000 | 47292 |
| 4 | 896.000 | 77717 |

\*\*\* CLASS-5000 \*\*\* Report No. = 1 Data : SIM11A.D11 111/05/2 701:22:3

Sample : sample 1

ID :

Sample Amount : 1

Dilution Factor : 1

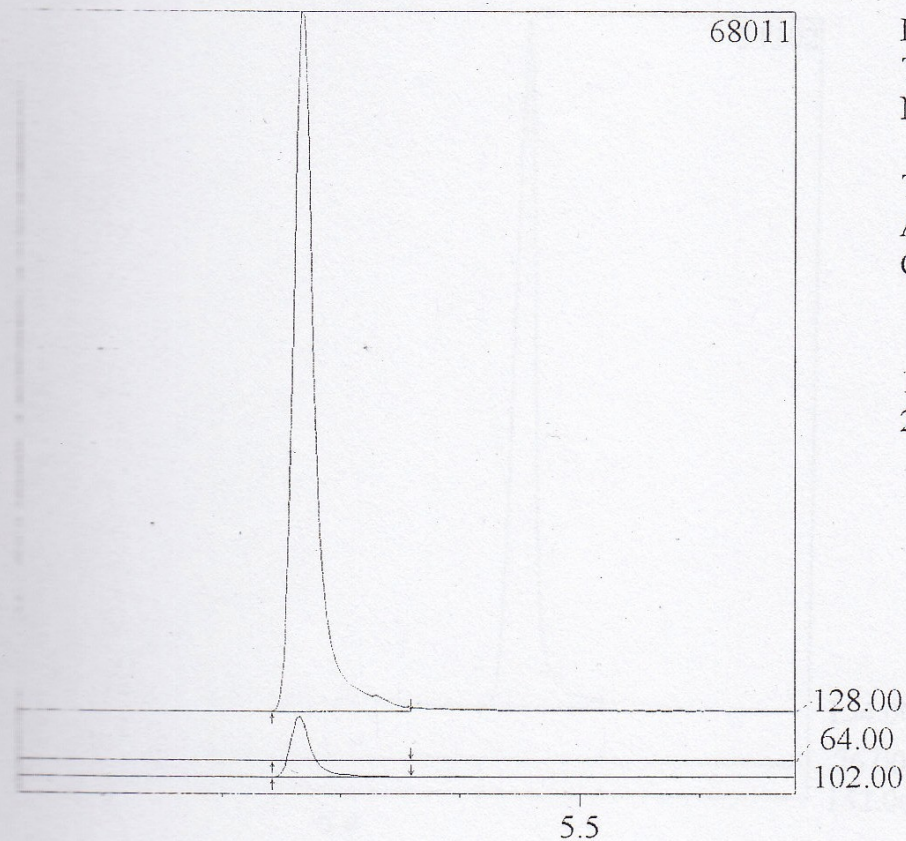
Type : Unknown

Operator :

Method File Name : SIM11A1.MET

Vial No. : 1

Barcode :



ID : 1 M/Z : 128.00

Type : Target

Name : naftaleno

Time : 5.267

Area : 86372

Conc. : 538.708ppb

|   | M/Z    | Area | %Rel.Int.to Target |
|---|--------|------|--------------------|
| 1 | 64.00  | 0    | 0                  |
| 2 | 102.00 | 7510 | 9                  |



\*\*\* CLASS-5000 \*\*\* Report No. = 1 Data : SIM11A.D11 111/05/2 701:22:3

Sample : sample 1

ID :

Sample Amount : 1

Dilution Factor : 1

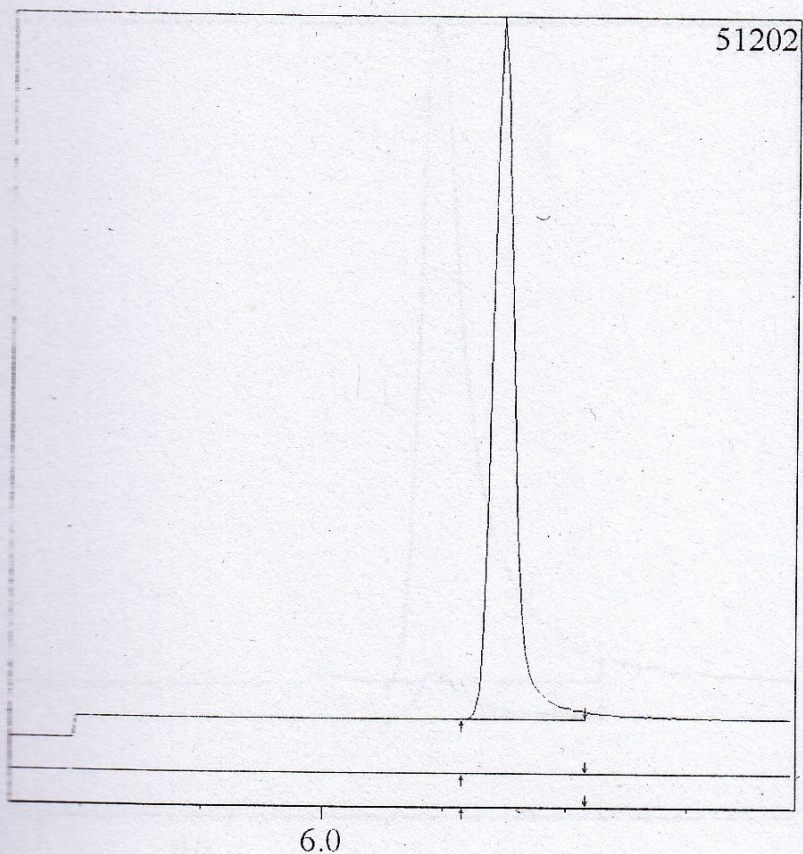
Type : Unknown

Operator :

Method File Name : SIM11A1.MET

Vial No. : 1

Barcode :



ID : 2 M/Z : 154.00

Type : Target

Name : bifenilo

Time : 6.147

Area : 59388

Conc. : 559.644ppb

|   | M/Z    | Area | %Rel.Int.to Target |
|---|--------|------|--------------------|
| 1 | 76.00  | 0    | 0                  |
| 2 | 152.00 | 0    | 0                  |



\*\*\* CLASS-5000 \*\*\* Report No. = 1 Data : SIM11A.D11 111/05/2 701:22:3

Sample : sample 1

ID :

Sample Amount : 1

Dilution Factor : 1

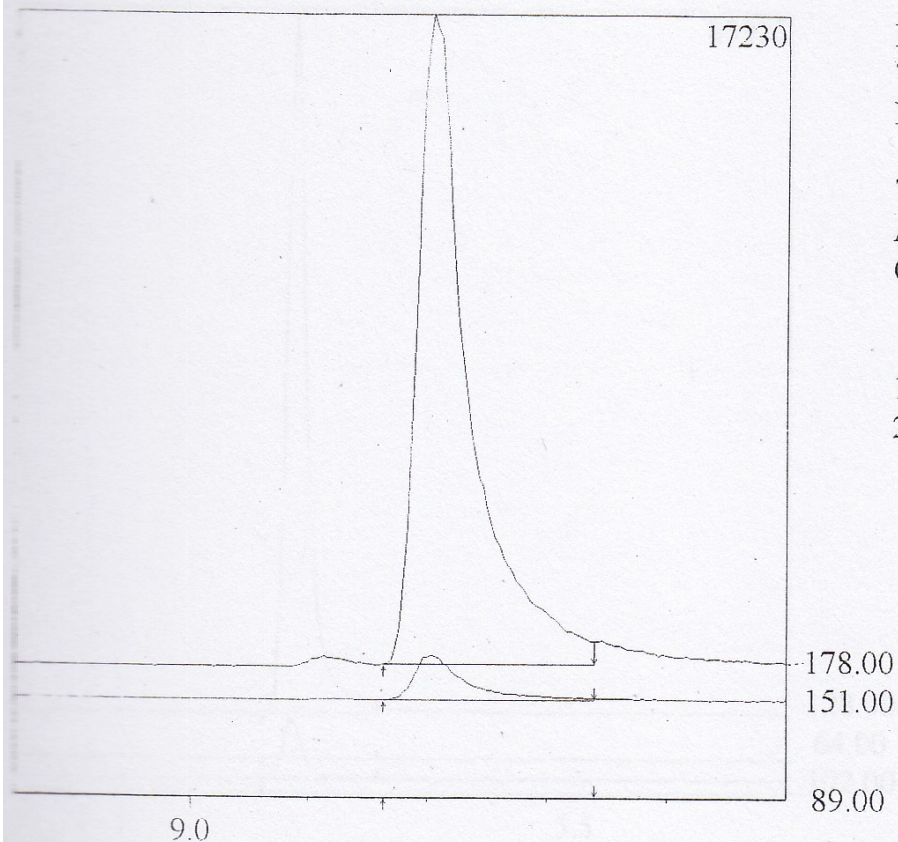
Type : Unknown

Operator :

Method File Name : SIM11A1.MET

Vial No. : 1

Barcode :



ID : 3 M/Z : 178.00

Type : Target

Name : antraceno

Time : 9.203

Area : 47497

Conc. : 594.122ppb

|   | M/Z    | Area | %Rel.Int.to Target |
|---|--------|------|--------------------|
| 1 | 151.00 | 3225 | 7                  |
| 2 | 89.00  | 0    | 0                  |

# REFERENCES

- Environment – Province of Columbia.  
<http://www.gov.bc.ca/env>
- Illinois Department of Public Health.  
<http://www.idph.state.il.us/envhealth>
- Practical sessions at Institute of Science and Technology of Mollet del Vallès, Catalonia - Spain?



Thank you  
for your attention !

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